FLORIDA DEPARTMENT OF TRANSPORTATION
State Materials Office
5007 NE 39th Avenue
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Florida Test Method
For
CARBONATES AND ORGANIC MATTER IN BASE MATERIALS

Designation: FM 5-514

1. SCOPE

This method covers the chemical analysis of cemented coquina and bank run shell carbonates of calcium and magnesium and organic matter such as wood trash or other vegetation.

Note 1: The values stated in SI units are to be regarded as the standard. The values given in parenthesis are for information only.

2. APPLICABLE DOCUMENTS

2.1 AASHTO Standard:

M 231 Specification for Weighing Devices in the Testing of Materials

2.2 ASTM Standard:

E 50 Recommended Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

2.3 Other Documents:

FM 1-T 248 Florida Test Method for Reducing Field Samples of Aggregate to Testing Size

FM 5-532 Florida Test Method for the Field Evaluation of Sealants for Shoulder Joints

MATERIALS SAFETY DATA SHEETS

3. OUTLINE OF METHOD

3.1 Impurities are determined as acid insoluble matter and as the ammonium hydroxide precipitate; carbonates are then calculated by difference. Organic matter is determined as the loss on ashing of acid insoluble matter.

4. APPARATUS

4.1 Bunsen burners with tripods or suitable hot plates.

4.2 Balance, analytical, sensitive to 0.1 mg complying with AASHTO M 231, Class A,

4.3 Beakers, 300-mL or other convenient size.

4.4 Burettes, dispensing, 50.00-mL capacity.

4.5 Crucibles, porcelain, 15-mL nominal capacity.

4.6 Desiccator with drierite or other efficient desiccant.

4.7 Drying oven, gravity, capable of maintaining a temperature of 105 to 115°C. (221 to 239°F)

4.8 Filter funnels, glass, 60° nominal, 75-mm nominal top diameter.

4.9 Gooch crucibles, 25-mL nominal capacity.

4.10 Jaw crusher and grinder capable of reducing dried samples to 90% passing the 425-μm (No. 40) sieve and with 100% passing the 2.00-mm (No. 10) sieve. Manual crushing of samples may be substituted for mechanical jaw crushers provided the integrity of the sample is maintained.

4.11 Muffle furnace capable of maintaining temperatures of 600 ± 50°C (1030 to 1200°F) and 900 ± 50°C (1550 to 1750°F).

4.12 Vacuum filter flask, 1-L capacity.

4.13 Stirring rods and rubber policemen.

4.14 Wash bottles, polyethylene squeeze type.

5. REAGENTS AND SUPPLIES

5.1 Hydrochloric acid,* 1:5. Add 1 volume of reagent grade hydrochloric acid to 5 volumes distilled or deionized water and mix.

5.2 Ammonium hydroxide,* 1:5. Add 1 volume of reagent grade ammonium hydroxide to 5 volumes distilled or deionized water and mix.

5.3 Asbestos fiber,* Gooch grade, medium fiber length. Glass microfiber filter discs (21 mm) such as Whatman No. 934-AH may be used in lieu of asbestos.

5.4 Filter paper, Whatman No. 41, or equal, 125 mm (Whatman No. 4 is an acceptable

equal.)

5.5 Phenolphthalein Indicator. Dissolve 1 g indicator grade phenolphthalein*in 100 mL reagent grade methanol* or denatured alcohol.*

Note 2 The concentrated acid and alkali should be handled under an efficient fume hood. The dilute (1:5) solutions may be used outside the hood without known harm. Avoid contact of all reagents with the skin and eyes. In the case of accidental splashes in the eyes, flush thoroughly with water and contact a physician.

6. SAMPLE PREPARATION (Use FM 1-T 248 where splitting or quartering is indicated).

6.1 If the material is damp when received from the field, it shall be dried until it becomes friable under the trowel. It may be air dried or by use of a drying apparatus provided the temperature does not exceed 60°C (140°F).

Note 3 Minimum gross sample size for Cemented Coquina, Shell Rock and Bank Run Shell materials shall be 23 kg (50 lb).

6.2 For materials to be used as base or stabilizers, the entire sample shall be passed through a crusher set at a maximum opening of 19 mm, with an under tolerance of 3 mm (3/4 in., with an under tolerance of 1/8 in.). Alternately, the entire sample may be crushed before the drying operation in Section 6.1.

6.3 Quarter to a convenient fraction - at least 1.4 kg (3 lb) for oven drying (at least 2.3 kg (5 lb) for Cemented Coquina, Shell Rock and Bank Run Shell).

6.4 Oven dry the split from Section 5.3 at 110° ± 5°C (230° ± 9°F) for 12 h minimum (overnight is convenient).

6.5 Recrush the split sample if needed to a size that will pass the pulverizer throat.

6.6 Split the recrushed material until a sample weighing at least 0.1 kg (1/4 lb.) or approx. 0.25 L (1/2 pt) is obtained. For Cemented Coquina, Shell Rock and Bank Run Shell at least 0.5 kg (1 lb) or approx. 1 L (1 qt) is required.

6.7 Pass the entire final sample through a pulverizer so that 90% passes 425-μm (No. 40) and 100% passes 2.00-mm (No. 10) sieve, and place in a moisture free container to assist analysis. This may require two passes through the pulverizer. For Cemented Coquina, Shell Rock and Bank Run Shell, split the pulverized material until a sample weighing at least 0.1 kg (1/4 lb) or approx. 0.25 L (1/2 pt.) is obtained.

6.8 Clean the crusher, pulverizer, and splitters between samples to avoid contamination of the next sample.

7. METHOD

7.1 Give the sample a final mixing by stirring with a spatula.

7.2 Weigh 1 g sample accurately and transfer to 300-mL beaker by brushing.

7.3 Add 20 mL of 1:5 hydrochloric acid* to beaker slowly.

7.4 Bring to a momentary boil over a Bunsen flame or hot plate. Allow to stand until the last of the gas has evolved from the sample particles.

7.5 Filter through tared Gooch ignited crucible, transferring all solid material in the beaker to the Gooch by a combination of scrubbing and washing with water. Wash down walls and bottom of Gooch crucible twice with water. Save filtrate (see Section 7.8).

Note 4 A tared Gooch crucible is prepared by pouring a water suspension of asbestos fiber *into the crucible and sucking it down to a pad thick enough to trap the sample particles on the filtering set-up. The crucible is then heated in a muffle furnace at 950 ± 50°C (1550 to 1750°F) for 30 min., cooled in a desiccator and weighed.

7.6 Dry Gooch crucible for 2 h at 105 to 115°C (221 to 239°F), cool in desiccator to room temperature and weigh. Increase in mass represents insoluble silica, clay, and organic matter (Residue A).

7.7 Ignite Gooch crucible at 600 ± 50°C (1030 to 1200°F) for 30 min, cool to room temperature in desiccator, and reweigh. Loss in mass represents organic matter. (Loss B).

7.8 Transfer filtrate from Section 7.5 back to original beaker, neutralize to faint pink with phenolphthalein *(2 drops) using 1:5 ammonium hydroxide*. Bring solution to momentary boil, then allow to cool until precipitate has settled enough for rapid filtration. Filter through No. 41 paper, transferring all the precipitate to paper by washing with water. Wash paper five times, allowing paper to drain between washings.

7.9 Transfer drained paper and contents to a porcelain crucible ash for 30 min at 900 ± 50°C (1550 to 1750°F). Cool crucible and contents in desiccator and weigh ash by transferring carefully to balance pan (Residue C).

7.10 Calculations:
% Carbonates of calcium and magnesium = (1.000 - Residues A and C) x 100

% Organic Matter = Loss B x 100.

8. SHORT METHOD FOR CARBONATES ONLY

8.1 Place 1.000 g in 300-mL beaker.

8.2 Add 20 mL of 1:5 hydrochloric acid*.

8.3 Heat to boiling.

8.4 Allow to stand until the last of the gas has evolved from the sample.

8.5 Add 3 drops of phenolphthalein solution*.

8.6 Titrate to pink color with 1:5 ammonium hydroxide* and boil momentarily.

8.7 Filter and wash well with water.

8.8 Ignite in porcelain crucible at 900 ± 50°C (1550 to 1750°F) (or bright red) until all carbon is destroyed (30 min or longer).

8.9 Cool and weigh contents of crucible as insoluble residue (R).

8.10 The carbonate content is calculated as follows:

$$C = \frac{(W - R)}{W} \times 100$$

Where:
C = % of carbonates of calcium and magnesium
W = mass of sample
R = insoluble residue

9. PRECISION

9.1 Cooperative tests have shown that these differences between labs are reasonable:

Carbonate Range	
90% and up	± 0.4%
50% - 75%	± 0.9%

10. SAFETY

10.1 Each hazardous material required in a procedure has been indicated by an asterisk indicating that the material safety data sheet should be consulted and cautions observed as indicated. Some judgment can be used. For example, dispensing 1 L of

concentrated ammonium hydroxide* from a 100-L carboy would indicate the need for a face shield; while dispensing eight drops from a 1-kg bottle would indicate that judgment could be used about the face shield as long as good ventilation was maintained.

Additionally, no employee is to use any of the hazardous reagents until thoroughly trained in the procedure and shown the proper use of devices such as hoods, ventilation fans, pipettes, etc.

Section 8 of ASTM Specification E 50 give additional helpful safety pointers.